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PCT

NOTICE INFORMING THE APPLICANT OF THE **COMMUNICATION OF THE INTERNATIONAL** APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

From the INTERNATIONAL BUREAU

IWAHASHI, Yuji 1-11-8, Higashikanagawa Kanagawa-ku, Yokohama-shi Kanagawa 221-0044 JAPON

Date of mailing (day/month/year)

28 December 2000 (28.12.00)

Applicant's or agent's file reference

SS1245

IMPORTANT NOTICE

International application No.

International filing date (day/month/year) 31 May 2000 (31.05.00)

Priority date (day/month/year)

PCT/JP00/03522

Applicant SHISEIDO CO., LTD. et al

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:

AG,AU,DZ,KP,KR,MZ,US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE,AL,AM,AP,AT,AZ,BA,BB,BG,BR,BY,CA,CH,CN,CR,CU,CZ,DE,DK,DM,EA,EE,EP,ES,FI,GB,GD, GE,GH,GM,HR,HU,ID,IL,IN,IS,JP,KE,KG,KZ,LC,LK,LR,LS,LT,LU,LV,MA,MD,MG,MK,MN,MW,MX, NO,NZ,OA,PL,PT,RO,RU,SD,SE,SG,SI,SK,SL,TJ,TM,TR,TT,TZ,UA,UG,UZ,VN,YU,ZA,ZW The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 28 December 2000 (28.12.00) under No. WO 00/78117

REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland

Authorized officer

J. Zahra

Facsimile No. (41-22) 740.14.35

Telephone No. (41-22) 338.83.38

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date 28 December 2000 (28.12.2000)

PCT

(10) International Publication Number WO 00/78117 A2

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English

(71) Applicants (for all designated States except US): SHI-SEIDO CO., LTD. [JP/JP]; 5-5, Ginza 7-chome, Chuo-ku, Tokyo 104-8010 (JP). SODA AROMATIC CO., LTD. [JP/JP]; 2-3, Nihonbashikodenmacho, Chuo-ku, Tokyo 103-8366 (JP).

(72) Inventors; and

(75) Inventors/Applicants (for US only): YOMOGIDA, Katsuyuki [JP/JP]; Shiseido Research Center(1), 1050. Nippa-cho, Kohoku-ku, Yokohama-shi, Kanagawa 223-8553 (JP). HIROSE, Seiichi [JP/JP]; Shiseido Research Center(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-shi, Kanagawa 223-8553 (JP). YOSHIMURA, Masanori [JP/JP]; Shiseido Research Center(1), 1050, Yokohama-shi, Nippa-cho, Kohoku-ku, Kanagawa 223-8553 (JP). HARIGAE, Hitoshi [JP/JP]; Soda Aromatic Co., Ltd., 2-3, Nihonbashikodenmacho, Chuo-ku, Tokyo 103-8366 (JP). KATAOKA, Masayuki [JP/JP]; Soda Aromatic Co., Ltd., 2-3, Nihonbashikodenmacho, Chuo-ku, Tokyo 103-8366 (JP).

(74) Agent: IWAHASHI, Yuji; 1-11-8, Higashikanagawa, Kanagawa-ku, Yokohama-shi, Kanagawa 221-0044 (JP).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

- Upon request of the applicant, before the expiration of the time limit referred to in Article 21(2)(a).
- Without international search report and to be republished upon receipt of that report.
- Without classification; title and abstract not checked by the International Searching Authority.

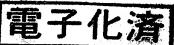
For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

17 A

(54) Title: METHOD OF COLLECTION OF FRAGRANCE INGREDIENT, PERFUME COMPOSITION CONTAINING THIS FRAGRANCE INGREDIENT AND COSMETICS CONTAINING THIS FRAGRANCE INGREDIENT

(57) Abstract: A method of collection of the fragrance ingredient of fragrant wood comprises extracting an essential oil ingredient by a solvent from the fragrant wood and collecting the fragrance ingredient in a vapor-phase by heating the extract, a perfume composition contains the fragrance ingredient, and a cosmetic contains the fragrance ingredient. The fragrance ingredient is obtained without a burnt smell, and it has an aroma of the fragrant wood.





PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

From the INTERNATIONAL BUREAU
To:

IWAHASHI, Yuki 1-11-8, Higashikanagawa Kanagawa-ku, Yokohama-shi Kanagawa 221-0044 JAPON

Date of mailing (day/month/year) 13 November 2000 (13.11.00)	7
Applicant's or agent's file reference SS1245	IMPORTANT NOTIFICATION
International application No. PCT/JP00/03522	International filing date (day/month/year) 31 May 2000 (31.05.00)
The following indications appeared on record concerning: X the applicant X the inventor	the agent
Name and Address 1) YOMOGIDA, Katsuyuki 2) HIROSE, Seiichi 3) YOSHIMURA, Masanori Shiseido Research Center(1)	State of Nationality State of Residence JP JP Telephone No.
1050, Nippa-cho, Kohoku-ku Yokohama-shi, Kanagawa 223-8553 Japan (applicants and inventors for all designated States)	Facsimile No. Teleprinter No.
The International Bureau hereby notifies the applicant that t X the person	
1) YOMOGIDA, Katsuyuki 2) HIROSE, Seiichi 3) YOSHIMURA, Masanori Shiseido Research Center(1) 1050, Nippa-cho, Kohoku-ku Yokohama-shi, Kanagawa 223-8553	JP JP Telephone No. Facsimile No.
Japan (applicants for US and inventors for all designated States)	Teleprinter No.
3. Further observations, if necessary:	
4. A copy of this notification has been sent to:	
X the receiving Office	the designated Offices concerned
the International Searching Authority the International Preliminary Examining Authority	the elected Offices concerned other:
The International Bureau of WIPO	Authorized officer

Telephone No.: (41-22) 338.83.38

Facsimile No.: (41-22) 740.14.35

1211 Geneva 20, Switzerland



	From the INTERNATIONAL BUREAU
PCT	То:
NOTIFICATION OF THE RECORDING OF A CHANGE (PCT Rule 92bis.1 and Administrative Instructions, Section 422) Date of mailing (day/month/year) 13 November 2000 (13.11.00)	IWAHASHI, Yuki 1-11-8, Higashikanagawa Kanagawa-ku, Yokohama-shi Kanagawa 221-0044 JAPON
Applicant's or agent's file reference	<u> </u>
SS1245	IMPORTANT NOTIFICATION
International application No.	International filing date (day/month/year)
PCT/JP00/03522	31 May 2000 (31.05.00)
The following indications appeared on record concerning: X the applicant X the inventor	the agent the common representative
Name and Address	State of Nationality State of Residence
1) HARIGAE, Hitoshi 2) KATAOKA, Masayuki	JP JP
Soda Aromatic Co., Ltd. 2-3, Nihonbashikodenmacho	Telephone No.
Chuo-ku, Tokyo 103-8366	Facsimile No.
Japan (applicants and inventors for all designated	
States)	Teleprinter No.
2. The International Bureau hereby notifies the applicant that	the following shapes has been recorded accounting.
X the person the name the ad	
Name and Address	State of Nationality State of Residence
1) HARIGAE, Hitoshi 2) KATAOKA, Masayuki	JP JP Telephone No.
Soda Aromatic Co., Ltd. 2-3, Nihonbashikodenmacho	relephone No.
Chuo-ku, Tokyo 103-8366 Japan	Facsimile No.
(applicants for US and inventors for all designated States)	
designated States)	Teleprinter No.
3. Further observations, if necessary:	
4. A copy of this notification has been sent to:	
X the receiving Office	the designated Offices concerned
the International Searching Authority	the elected Offices concerned
the International Preliminary Examining Authority	other:
The leavest 10 course	Authorized officer
The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Shinji IGARASHI

Telephone No.: (41-22) 338.83.38

Facsimile No.: (41-22) 740.14.35

PCT

For receiving Office use only International Application No. International Filing Date Name of receiving Office and "PCT International Application"

REQUEST The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty. Applicant's or agent's file reference SS1245 (if desired) (12 characters maximum) TITLE OF INVENTION Box No. I METHOD OF COLLECTION OF FRAGRANCE INGREDIENT, PERFUME COMPOSITION CONTAINING THIS FRAGRANCE INGREDIENT AND COSMETICS CONTAINING THIS FRAGRANCE INGREDIENT **APPLICANT** Box No. II (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) Name and address: This person is also inventor. SHISEIDO CO., LTD. Telephone No. 03-3572-5111 5-5, Ginza 7-chome, Chuo-ku, Facsimile No. Tokyo 104-8010 JAPAN Teleprinter No. State (i.e. country) of nationality: State (i.e. country) of residence: **JAPAN JAPAN** all designated States except the United States of America the United States of America only This person is applicant the States indicated in all designated the Supplemental Box for the purposes of: FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S) Box No. III (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) Name and address: This person is: applicant only SODA AROMATIC CO., LTD. applicant and inventor Nihonbashikodenmacho, Chuo-ku, 2-3, Tokyo 103-8366 JAPAN inventor only (If this check-box is marked, do not fill in below.) State (i.e. country) of nationality: State (i.e. country) of residence: **JAPAN JAPAN** the States indicated in the Supplemental Box the United States of America only This person is applicant all designated all designated States except the United States of America for the purposes of: Further applicants and/or (further) inventors are indicated on a continuation sheet. AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE Box No. IV The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: agent common representative (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) Telephone No. Name and address: 045-453-4316 Patent Attorney (Reg. No. 9290) Facsimile No. IWAHASHI Yuji 045-453-4317 1-11-8, Higashikanagawa, Kanagawa-ku, Yokohama-shi, Teleprinter No. Kanagawa 221-0044 JAPAN Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to

indicate a special address to which correspondence should be sent.

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Sheet	No.		_	

Continuation of Box No. III FURTHER APPLICANTS AN	D/OR (FURTHER) INV	ENTORS
If none of the following sub-boxes is used, t	his sheet is not to be incli	uded in the request.
Name and address: (Family name followed by given name; for a designation. The address must include postal coa	legal entity, full official de and name of country.)	This person is:
YOMOGIDA Katsuyuki	_	applicant only
c/o SHISEIDO RESEARCH CENTER(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-s Kanagawa 223-8553 JAPAN	shi,	applicant and inventor inventor only (If this check-box is marked, do not fill in below.)
State (i.e. country) of nationality:	State (i.e. country) of res	sidence:
JAPAN	*	JAPAN
This person is applicant all designated all designated for the purposes of:	States except the of America of America	United States America only the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a designation. The address must include postal code HIROSE Seiichi c/o SHISEIDO RESEARCH CENTER(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-s Kanagawa 223-8553 JAPAN		This person is: applicant only papplicant and inventor inventor only (If this check-box is marked, do not fill in below.)
State (i.e. country) of nationality: JAPAN	State (i.e. country) of res	JAPAN
This person is applicant for the purposes of: all designated the United States all designated		United States America only the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a designation. The address must include postal code of the control	ie ana name oj country.j	This person is: applicant only applicant and inventor inventor only (If this check-box is marked, do not fill in below.)
State (i.e. country) of nationality:	State (i.e. country) of re-	sidence:
JAPAN		JAPAN
This person is applicant for the purposes of: All designated all designated the United States	States except the of America of A	United States America only the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a designation: The address must include postal contact the second sec	legal entity, full official de and name of country.)	This person is: applicant only applicant and inventor inventor only (If this check-box is marked, do not fill in below.)
State (i.e. country) of nationality: JAPAN	State (i.e. country) of res	sidence: JAPAN
This person is applicant all designated for the purposes of:		e United States the States indicated in the Supplemental Box
Form PCT/RO/101 (continuation sheet) (July 1993; reprint Januar		eet. See Notes to the request form

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Continuation of Box No. III FURTHER APPLICANTS AND/OR (FURTHER)	INVENTORS
If none of the following sub-boxes is used, this sheet is not to be	included in the request.
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	This person is:
KATAOKA Masayuki	applicant only
c/o SODA AROMATIC CO., LTD. 2-3, Nihonbashikodenmacho , Chuo-ku,	applicant and inventor
Tokyo 103-8366 JAPAN	inventor only (If this check-box is marked, do not fill in below.)
State (i.e. country) of nationality: State (i.e. country) of	f residence:
JAPAN	JAPAN
This person is applicant all designated all designated States except the United States of America	the United States of America only the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	This person is:
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State (i.e. country) of nationality: State (i.e. country) of	f residence:
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This person is applicant for the purposes of: all designated the United States except the United States of America	the United States of America only the Supplemental Box
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)	This person is:
•	applicant only
	applicant and inventor
	inventor only (If this check-box is marked, do not fill in below.)
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This person is applicant all designated all designated States except the United States of America	the United States of America only the States indicated in the Supplemental Box
Name and address: (Family name followed by given name; for a legal entity, full official designation: The address must include postal code and name of country.)	This person is:
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	inventor only (If this check-box
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This person is applicant for the purposes of: all designated all designated States except the United States of America	the United States of America only the Supplemental Box
Further applicants and/or (further) inventors are indicated on another continuation	n sheet.

Box No	o.V DESIGNATION OF STATES		
The fol	llowing designations are hereby made under Rule 4.9(a)	(mark the d	applicable check-boxes; at least one must be marked):
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—/	Republic of Korea	Check-	boxes reserved for designating States which have party to the PCT after issuance of this sheet:
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☑ LK	Sri Lanka	A (/c	1. THUJUN AND PHIDHOUND BY DELIZE
			de above, the applicant also makes under Rule 4.9(b) all other
			tion(s) indicated in the Supplemental Box as being excluded
trom the	e scope of this statement. The applicant declares that the	iose addit the from t	ional designations are subject to confirmation and that any he priority date is to be regarded as withdrawn by the applicant
at the ex	piration of that time limit. (Confirmation (including fees) mus	st reach the	e receiving Office within the 15-month time limit.)

Sheet No. ..5...

Box No. VI PRIORITY CI	AIM Fu	rther priority claims are indicated in th	e Supplemental Box
The priority of the following ea	rlier application(s) is hereby claime	d:	
Country (in which, or for which, the application was filed)	Filing Date (day/month/year)	Application No.	Office of filing (only for regional or international application)
item (1)			
item (2)			
item (3)			
The receiving Office is he Bureau a certified copy of	ertified copy of the earlier application is a fee may be required): ereby requested to prepare and transfithe earlier application(s) identified NAL SEARCHING AUTHORITY	above as item(s):	oses of the present international
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(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau





(43) International Publication Date 28 December 2000 (28.12.2000)

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(10) International Publication Number WO 00/78117 A3

(51) International Patent Classification7:

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Kanagawa-ku, Yokohama-shi, Kanagawa 221-0044 (JP).

- (21) International Application Number:
 - PCT/JP00/03522

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- (25) Filing Language:

English

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- (71) Applicants (for all designated States except US): SHI-SEIDO CO., LTD. [JP/JP]; 5-5, Ginza 7-chome, Chuo-ku, Tokyo 104-8010 (JP). SODA AROMATIC CO., LTD. [JP/JP]; 2-3, Nihonbashikodenmacho, Chuo-ku, Tokyo 103-8366 (JP).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): YOMOGIDA, Katsuyuki [JP/JP]; Shiseido Research Center(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-shi, Kanagawa 223-8553 (JP), HIROSE, Seiichi [JP/JP]; Shiseido Research Center(1), 1050, Nippa-cho, Kohoku-ku, Yokohama-shi, Kanagawa 223-8553 (JP). YOSHIMURA, Masanori [JP/JP]; Shiseido Research Center(1), 1050, Kohoku-ku, Yokohama-shi, Kanagawa Nippa-cho, 223-8553 (JP). HARIGAE, Hitoshi [JP/JP]; Soda Aromatic Co., Ltd., 2-3, Nihonbashikodenmacho, Chuo-ku, Tokyo 103-8366 (JP). KATAOKA, Masayuki [JP/JP]; Soda Aromatic Co., Ltd., 2-3, Nihonbashikodenmacho, Chuo-ku, Tokyo 103-8366 (JP).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(74) Agent: IWAHASHI, Yuji; 1-11-8, Higashikanagawa,

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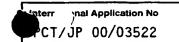


(54) Title: METHOD OF COLLECTION OF FRAGRANCE INGREDIENT, PERFUME COMPOSITION AND COSMETICS CONTAINING IT

(57) Abstract: A method of collection of the fragrance ingredient of fragrant wood comprises extracting an essential oil ingredient by a solvent from the fragrant wood and collecting the fragrance ingredient in a vapor-phase by heating the extract, a perfume composition contains the fragrance ingredient, and a cosmetic contains the fragrance ingredient. The fragrance ingredient is obtained without a burnt smell, and it has an aroma of the fragrant wood.

INTERNATIONAL SEARCH REPORT





A. CLASSI IPC 7	FICATION OF SUBJECT MATTER C11B9/02		
According to	o International Patent Classification (IPC) or to both national classifica	ation and IPC	·
B. FIELDS	SEARCHED		
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Documentat	tion searched other than minimum documentation to the extent that s	uch documents are included in the fields se	earched
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C. DOCUMI	ENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the rel	evant passages	Relevant to claim No.
Υ	DATABASE WPI Section Ch, Week 199418		1-11
	Derwent Publications Ltd., Londor Class D23, AN 1994-148156	n, GB;	
	XP002156947 & JP 06 093287 A (KANEBO LTD),		
	5 April 1994 (1994-04-05) cited in the application		
	abstract		
Υ	DE 195 32 017 A (LENTZ HARRO)		1-11
	6 March 1997 (1997-03-06) the whole document		
Y	US 4 920 096 A (BEDOUKIAN PAUL Z) 24 April 1990 (1990-04-24)	•	1,3-11
	the whole document		
Furth	ner documents are listed in the continuation of box C.	Palent family members are listed	in annex.
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Patent document cited in search report		Publication date	Patent family member(s)	Publication date
JP 6093287	Α	05-04-1994	NONE	
DE 19532017	Α	06-03-1997	NONE	
US 4920096	A	24-04-1990	NONE	·

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(54) Title: METHOD OF COLLECTION OF FRAGRANCE INGREDIENT, PERFUME COMPOSITION CONTAINING THIS FRAGRANCE INGREDIENT AND COSMETICS CONTAINING THIS FRAGRANCE INGREDIENT

(57) Abstract: A method of collection of the fragrance ingredient of fragrant wood comprises extracting an essential oil ingredient by a solvent from the fragrant wood and collecting the fragrance ingredient in a vapor-phase by heating the extract, a perfume composition contains the fragrance ingredient, and a cosmetic contains the fragrance ingredient. The fragrance ingredient is obtained without a burnt smell, and it has an aroma of the fragrant wood.

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DESCRIPTION

METHOD OF COLLECTION OF FRAGRANCE INGREDIENT, PERFUME COMPOSITION CONTAINING THIS FRAGRANCE INGREDIENT AND COSMETICS CONTAINING THIS FRAGRANCE INGREDIENT

Technical Field

The present invention relates to an improved method of collection a fragrance ingredient and a perfume composition containing the fragrance ingredient and a cosmetic containing the fragrance ingredient. The invention provides an improvement of the fragrance quality of a perfume composition containing the fragrance ingredient. Also, the invention provides an improvement of the fragrance quality of a cosmetic containing the fragrance ingredient.

Background Art

An agarwood is the fragrant wood containing the resin that has unique aroma. The producing district of agarwood is found mainly in Southeast Asia. The fragrance of the agarwood obtained by heating or burning was used as incense. Especially among the agarwood, the high quality agarwood called "Kyara", is highly prized.

Conventionally, the collection method of fragrance ingredient from a fragrant wood is by the thermal decomposion of the fragrant wood powder with a mixed gas as described in Japanese Unexamined Patent Publication No. Hei 6-93287. However, this collection method had a disadvantage that a burnt smell depending on the dry distillation of fragrant wood can not be removed. Also, this method is a complicated method that requires a special apparatus and an adjustment of heating temperature and mixed gas. Also, the decomposition product of fragrant wood yields ingredient other than original fragrance ingredient and the colored solution. Therefore, it was impossible to reproduce the original aroma of agarwood completely from the collected fragrance ingredient of the prior art.

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Disclosure of Invention

An object of the present invention is to provide a method that collects only desirable fragrance ingredient effectively without a burnt smell. It is an object to reproduce the original aroma of fragrant wood from fragrant wood. It is further object of the present invention is to provide an application of the original aroma fragrance ingredient to the various fragrance products and cosmetics.

As a result of diligent study by the present inventors, the present invention has been accomplished. The present inventors have found that use of solvent extraction is able to achieve the above objects.

A method of collection of a fragrance ingredient of the present invention comprises extracting the essential oil ingredient by solvent from a fragrant wood and collecting the fragrance ingredient in vapor-phase by heating the extract.

Also, in the method of collection of the fragrance ingredient of the present invention, it is preferable that said fragrance ingredient is discharged forcibly from a heating container by introducing an inert gas.

Also, in the method of collection of the fragrance ingredient of the present invention, it is preferable that said fragrance ingredient is collected to an absorption solvent and get a resultant as a fragrance collection liquid.

Also, in the method of collection of the fragrance ingredient of the present invention, it is preferable that said fragrant wood is agarwood.

Also, a perfume composition of the present invention contains said fragrance ingredient by means of the method of collection of the fragrance ingredient.

Also, in the perfume composition of the present invention, it is preferable that said perfume composition contains a fragrant wood extract.

Also, in the perfume composition of the present invention, it is preferable that a ratio of the fragrance collection solution/said fragrant wood extract is 0.25 to 9.

Also, in the perfume composition of the present invention, it is preferable that said perfume composition has an aroma of agarwood.

Also, a cosmetic of the present invention contains said fragrance ingredient by

means of the method of collection of the fragrance ingredient.

Also, in the cosmetic of the present invention, it is preferable that said cosmetic contains a fragrant wood extract.

Also, in the cosmetic of the present invention, it is preferable that a ratio of the fragrance collection liquid/said fragrant wood extract is 0.25 to 9.

Brief Description of Drawings

FIG.1 shows one example of the fragrance collection apparatus that is used for the present invention.

Best Mode for Carrying Out the Invention

The following is the explanation of the details for the collection method of a fragrance ingredient of the present invention.

Powder or chips of fragrant wood is processed by heating agitation extraction or by soaking extraction for extracting the essential oil ingredient. After filtering, the extract is obtained by removing the solvent. This extract is heated and the volatized fragrance in the head space is led outside the system. The introduction of inert gas is more suitable. The fragrance ingredient is collected in an absorption solvent which becomes a fragrance collection liquid. By this method, a fragrance ingredient that reproduces the original fragrance of fragrant wood is obtained. Also, the perfume composition containing this fragrance collection liquid can reproduce the original fragrance of the fragrant wood. Also, the cosmetic containing this fragrance collection liquid can improve the quality of aroma.

Extraction of Fragrance Ingredient

In the present invention, the powder or the pieces of the fragrant wood can be used suitably as a fragrant wood material. The fragrant wood in the present invention is a agarwood or a sandalwood etc. The agarwood is grouped into 6 kinds which is called "Rikkoku".

"Rikkoku" is derived from producing district of the fragrant wood, i.e.

"Kyara", "Rakoku", "Manaban", "Manaka", "Sasora", "Sumotara" etc. Many of the fragrant woods are produced from the hinterland of Vietnam, Cambodia and Myanmar. However, the differentiation of a producing district becomes meaningless now. The quality of tree is decided on the basis of the figure and aroma of fragrant wood.

A solvent is used to extract the essential oil ingredient containing the fragrance ingredient in the fragrant wood in the present invention. Water, methanol, ethanol, isopropanol, diethylether, pentane, hexane, propyleneglycol, glycerin etc. or these mixture solvent etc. are used for the solvent that extracts the essential oil ingredient containing the fragrance ingredient of the fragrant wood.

In the present invention, addition of the solvent for the extraction of 10 to 100 weight times to fragrant wood is desirable. Addition of the solvent for the extraction of 20 to 70 weight times to fragrant wood is more desirable. By this solvent extraction, the essential oil ingredient containing fragrance ingredient is extracted. As for extraction method, by the method of the public knowledge such as the heating extraction with agitation or the soaking extraction processing by using the solvent, the essential oil ingredient containing fragrance ingredient can be extracted.

A desirable process that extracts the essential oil ingredient which contains ingredients from fragrant wood is, for example, the extraction processing in 3 to 6 hours at 40° C to 60° C using heating extraction with agitation. Also, for example, it is desirable to extract for 1 to 10 days at room temperature using soaking extraction. Next, the crude extract (the essential oil ingredient) that is obtained is treated with solid-liquid separation by the optional method such as filtration or centrifugation. Furthermore the separated liquid that is obtained is concentrated and the extract is obtained. As for the concentration method of the separated liquid, for example, the method of public knowledge can be used, such as distilling and removing solvent under reduced pressure by the rotary evaporator.

Collection of fragrance ingredient

Next, the concentration extract that is obtained by the above described process is inserted into the fragrance collection apparatus. The fragrance ingredient volatilized by heating is discharge forcibly through collection apparatus by the aeration of an inert gas, and is collected to the absorption solvent inside the collection apparatus.

FIG.1 shows one example of the fragrance collection apparatus that is used to the collection method of the fragrance ingredient of the present invention. In FIG.1, the concentrated extraction liquid is heated in the heating container 1 together with an inert gas introduced through introduction pipe 2. The fragrance ingredient volatilizes in heating container 1. Next, the fragrance ingredient with the inert gas that is introduced from introduction pipe 2 which passes through the connection pipe 3, is introduced to the collection apparatus 4, and is collected in absorption solvent 5. In the present invention, an agitation apparatus can be attached to the heating container 1. Also, by using a drum style container or a flask style container as the heating container, it can also be heated with rotation. The collection apparatus can use either countercurrent contact style or batch style. In the case of batch style, it is desirable to connect 2 to 3 collection apparatuses to improve collection efficiency. Also it is desirable to cool the absorption solvent with ice water etc. to improve collection efficiency. Also, to increase gas-liquid interface, it is possible to make the tip part of the introduction pipe perforated pipe style. Also it is possible to destroy or make small bubles physically by agitation apparatus etc.

The heating temperature of the above-mentioned heating container 1 is preferably in the ranges of 150℃ to 200℃. Nitrogen, argon and carbon dioxide etc. gas used for inert gas. A gas flow rate for 1 g of extract oil is preferably the range of 20 ml/min to 200 ml/min. Also, according to the type of use of the collection liquid, methanol, ethanol, isopropanol, diethyl ether, pentane, hexane, hydrated methanol, hydrated ethanol, propylene glycol, dipropylene glycol etc. are used as absorption solvent. The amount of absorption solvent is preferably 10 to 100 times the amount of said extract. Also, preferably the absorption solvent is cooled. The preferable cooling temperature is 0 to 10℃.

Perfume composition

The fragrance ingredient that was collected in this way is preserved as the fragrance collection liquid which contains the fragrance ingredient. A perfume composition can contain the fragrance collection liquid combined with another perfume in optional proportion. By adding an extract of the fragrant wood to the perfume composition of the present invention, an improved more excellent aroma of fragrant wood can be reproduced. The manufacturing method of extract of the fragrant wood is sufficient with the method of public knowledge. A ratio by weight of fragrance collection liquid/fragrant wood extract is preferably approximately 0.25 to 9. In the case that this ratio missed, the original fragrance of fragrant wood may be damaged.

Also, the perfume composition in the present invention includes any product that is the perfume fragrance with except for food mainly, for example perfume, colon, bath agents, various kinds cleaners, sanitation articles, incense sticks, aromatics etc.

Cosmetics

The fragrance ingredient of the present invention can be added to the cosmetics and the like. The cosmetics of the present invention are, for example perfume, colon, foundation, lotion, cosmetic cream, cosmetic gel, hair dressing etc.; toiletry products such as soap, detergent, shampoo, rinse etc. The cosmetics of the present invention are the product that can contain fragrance in the perfume other than food mainly.

By adding an extract of the fragrant wood to the perfume composition of the present invention, an improved more excellent aroma of fragrant wood can be obtained. The manufacturing method of extract of the fragrant wood is sufficient with the method of public knowledge. A ratio by weight of fragrance collection liquid/fragrant wood extract is preferably approximately 0.25 to 9. In the case that this ratio missed, the original fragrance of fragrant wood may be damaged.

The following is the detailed explanation of the present invention by working examples.

Collection method of fragrance ingredient

(Working example 1)

10g of powder of fragrant wood "Kyara" was placed into 500 ml of Erlenmeyer flask with a plug and was extracted with 300g of diethyl ether for 10 days at room temperature. Then, the wood powder was separated from extract liquid by filtration. Diethyl ether was removed by evaporator under reduced pressure.

Next, the extract oil was transferred to Claisen flask type distillation apparatus, was heated to 180°C with an oil bath. Nitrogen gas was introduced with flow rate of 300 ml/min. The fragrance ingredient was collected for 60 minutes. In the collection method, 30ml of 95%ethanol was used as absorption solvent. The distilled fragrance ingredient with the gas was collected in the absorption solvent by a introducing tube whose exit was in the absorption solvent. The collection apparatus was connected in series (30ml×2) and the fragrance ingredient volatilized was collected. Considering increasing collection efficiency, the absorption solvent was cooled with an ice water bath.

(Working example 2)

The solvent that extracts fragrant wood was changed into methanol, and the extraction processing of the method similar to Working example 1 was carried out. Next, in the condition similar to Working example 1, the extract oil was transferred to Claisen flask type distillation apparatus, was heated to 180° C with an oil bath. Nitrogen gas was introduced with flow rate of 300 ml/min. The fragrance ingredient was collected for 60 minutes. In the collection method, 30ml of 95%ethanol was used as absorption solvent. The distilled fragrance ingredient with the gas was collected in the absorption solvent by a introducing tube whose exit was in the absorption solvent. The collection apparatus was connected in series $(30\text{ml} \times 2)$ and the fragrance ingredient volatilized was collected. Considering increasing collection efficiency, the absorption solvent was cooled with an ice water bath.

(Working example 3)

The solvent that extracts fragrant wood was changed into 95%ethanol, and the

extract was obtained by removing the solvent. Next, by the condition similar to Working example 1, the extract oil was transferred to Claisen flask type distillation apparatus, was heated to 180°C with an oil bath. Nitrogen gas was introduced with flow rate of 300 ml/min. The fragrance ingredient was collected for 60 minutes. In the collection method, 30ml of 95%ethanol was used as absorption solvent. The distilled fragrance ingredient with the gas was collected in the absorption solvent by a introducing tube whose exit was in the absorption solvent. The collection apparatus was connected in series (30ml×2) and the fragrance ingredient volatilized was collected. Considering increasing collection efficiency, the absorption solvent was cooled with an ice water bath.

(Working example 4)

The solvent that extracts fragrant wood was changed into hexane, and the extraction processing of the condition similar to Working example 1 was carried out and the extract was obtained by removing the solvent. Next, by the condition similar to Working example 1, the extract oil was transferred to Claisen flask type distillation apparatus, was heated to 180° C with an oil bath. Nitrogen gas was introduced with flow rate of 300 ml/min. The fragrance ingredient was collected for 60 minutes. In the collection method, 30ml of 95%ethanol was used as absorption solvent. The distilled fragrance ingredient with the gas was collected in the absorption solvent by a introducing tube whose exit was in the absorption solvent. The collection apparatus was connected in series $(30\text{ml} \times 2)$ and the fragrance ingredient volatilized was collected. Considering increasing collection efficiency, the absorption solvent was cooled with an ice water bath.

(Working example 5)

By the method similar to Working example 1, the extraction processing of fragrant wood was carried out with diethyl ether and the extract was obtained by removing the solvent.. Next, by the condition similar to Working example 1, the extract oil was transferred to Claisen flask type distillation apparatus, was heated to 150°C with an oil bath. Nitrogen gas

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was introduced with flow rate of 300 ml/min. The fragrance ingredient was collected for 60 minutes. In the collection method, 30ml of 95%ethanol was used as absorption solvent. The distilled fragrance ingredient with the gas was collected in the absorption solvent by a introducing tube whose exit was in the absorption solvent. The collection apparatus was connected in series $(30\text{ml} \times 2)$ and the fragrance ingredient volatilized was collected. Considering increasing collection efficiency, the absorption solvent was cooled with an ice water bath.

(Working example 6)

By the method similar to Working example 1, the extraction processing of fragrant wood was carried out with diethyl ether and the extract was obtained by removing the solvent. Next, by the condition similar to Working example 1, the extract oil was transferred to Claisen flask type distillation apparatus, was heated to 200° C with an oil bath. Nitrogen gas was introduced with flow rate of 300 ml/min. The fragrance ingredient was collected for 60 minutes. In the collection method, 30ml of 95%ethanol was used as absorption solvent. The distilled fragrance ingredient with the gas was collected in the absorption solvent by a introducing tube whose exit was in the absorption solvent. The collection apparatus was connected in series (30ml \times 2) and the fragrance ingredient volatilized was collected. Considering increasing collection efficiency, the absorption solvent was cooled with an ice water bath.

(Comparative example 1)

An dry distillated collection liquid was obtained from the fragrant wood powder by dry distillation with the apparatus similar to Working example 1. Next, the extract oil was transferred to Claisen flask type distillation apparatus, and it was heated to 180°C with an oil bath. Nitrogen gas was introduced with flow rate of 300 ml/min. The fragrance ingredient was collected for 60 minutes. In the collection method, 30ml of 95%ethanol was used as absorption solvent. The distilled fragrance ingredient with the gas was collected in the absorption solvent by an introducing tube whose exit was in the absorption solvent. The collection apparatus was connected in series (30ml × 2) and the fragrance ingredient

volatilized was collected. Considering increasing collection efficiency, the absorption solvent was cooled with an ice water bath. The dry distillation collection liquid that was collected was compared with Working example 1 of the present invention method.

Table 1 and Table 2 show the result of the extraction process with regard to Working examples 1 to 6 and Comparative example 1.

Table 1

Extraction condition				
	Extraction	solvent	Fragrant wood	Extraction
	Kind	Amount	Amount	time
Comp. Ex. 1	-		10g	_
Work. Ex. 1	Diethyl ether	300g	10g	7 days
Work. Ex. 2	Methanol	300g	10g	7 days
Work. Ex. 3	95%Ethanol	300g	10g	7 days
Work. Ex. 4	Hexane	300g	10g	7 days
Work. Ex. 5	Diethyl ether	300g	10g	7 days
Work. Ex. 6	Diethyl ether	300g	10g	7 days

Table 2

	Extracti	on result
	Extract amount	Coloring
Comp. Ex. 1		
Work. Ex. 1	2.67g	Liver brown
Work. Ex. 2	2.44g	Light-brown
Work. Ex. 3	2.51g	Light-brown
Work. Ex. 4	2.78g	Dark brown
Work. Ex. 5	2.58g	Liver brown
Work. Ex. 6	2.60g	Liver brown

Table 3 shows the parameters of the methods of the heating fragrance collection process with regard to Working examples 1 to 6 and Comparative example 1.

Table 3

	Gas	Fragra Flow Rate	nce ingre Temp.	dient collection Collection So Kind	Collection Time	
Comp. Ex. 1	Nitrogen	300ml	180°C	95%Ethanol	100g	60 min
Work. Ex. 1	Nitrogen	300ml	180℃	95% Ethanol	100g	60 min
Work. Ex. 2	Nitrogen	300ml	180℃	95% Ethanol	100g	60 min
Work. Ex.3	Nitrogen	300ml	180℃	95% Ethanol	100g	60 min
Work. Ex.4	Nitrogen	300ml	180℃	95% Ethanol	100g	60 min
Work. Ex.5	Nitrogen	300ml	150℃	95% Ethanol	100g	60 min
Work. Ex.6	Nitrogen	300ml	200℃	95% Ethanol	100g	60 min

Table 4 shows the evaluation of aroma with regard to Working examples 1 to 6 and Comparative example 1.

Table 4

Comp. Ex. 1 Colorless Woody aroma with burnt smell Work. Ex. 1 Colorless Woody aroma with density Work. Ex. 2 Colorless Woody aroma with mellowness	smell
Work. Ex. 1 Colorless Woody aroma with density Work. Ex. 2 Colorless Woody aroma with mellowness	
Work. Ex. 2 Colorless Woody aroma with mellowness	Yes
Work, Ex. 2 Coloness Woody aroma with mellowness	No
Work Ex 3 Colorless Woody aroma with mellowness	No
	No
Work Ex. 4 Colorless Woody aroma with slight quietude	No
Work. Ex 5 Colorless Woody aroma with density	No
	No

^{*} The total of absorption liquid in two trap was evaluated regarding aroma.

In Table 4, it is understood that the collection liquid of Comparative example 1 by dry distillation without extraction has a big fault that there is a burnt smell which is not desired as perfume material, although there is also woody aroma. As the result of Working examples 1 to 6, it is shown that no burnt smell exists at all, although there is a different aroma depending on kind of the extract solvent. Also, because the burnt smell is not collected from the head space from the heating to 250°C, most of the extracted ingredients are presumed to be oily ingredients. The extracted oil showed fairly deep brown to light-brown color in the extracts of any solvent (see Table 2). However, the fragrance collection liquid of the present invention was estimated to be colorless (see Table 4), which means that the part of the aroma is only collected.

Perfume composition

As the example of the perfume composition containing the fragrance collection liquid that was obtained from fragrant wood, the perfume composition for incense stick is shown in Table 5. Also, the result of the evaluation of the smell quality in the sensing test is shown in Table 5, which was evaluated. The evaluation standard is as follows. The evaluated values are average of 20 panels of specialist.

(Evaluation standard)

- 3: The smell quality with the aroma of fragrant wood was excellent
- 2: The smell quality with the aroma of fragrant wood was excellent slightly
- 1: The smell quality with the aroma of fragrant wood was inferior slightly
- 0: The smell quality with the aroma of fragrant wood was inferior considerably

Table 5

No.	Perfume material	Comp. Ex.2	Work. Ex.7	Comp Ex.3
1	Benzyl cinnamate	13	13	13
2	Raspberry Ketone	4	4	4
3	Vanillin	7	7	7
4	Ethyl vanillin	3	3	3
5	Coumarin	40	40	40
6	Vetiveryl acetate	3	3	3
7	Sandalwood oil	60	60	60
8	Ambroxan	3	3	3
9	Ethyl cinnamate	15	15	15
10	Methyl cinnamate	10	10	10
11	Cinnamic aldehyde	10	10	10
12	Phenylethyl alcohol	250	250	250

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13	Benzyl alcohol	45	45	45	
14	Benzaldehyde	7	7	7	
15	Bergamotal	20	20	20	
16	Fragrance collection liquid		10	_	
17	Dry distillation collection liquid	_	_	10	
Total	(by weight)	490	500	500	
Evaluation			3	0	
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In Table 5, it is understood that the perfume composition of Working example 7 containing the fragrant wood fragrance collection liquid obtained by the method of the present invention from fragrant wood has excellent agarwood aroma. On the other hand, Comparative example 3 containing dry distillation collection liquid had nasty and burnt smell, which degraded the smell quality. Also, in the comparison of between the aroma of Working example 7 and Comparative example 3, a higher-class impression was recognized in Working example 7 than Comparative example 3. The aroma of Working example 7 had a deep woody aroma which derives more natural feeling.

Next, as the example of the perfume composition containing the fragrance collection liquid that was obtained from fragrant wood, the perfume composition for the cosmetics is shown in Table 6. The present inventors tried to add the fragrant wood extract for reproducing better agarwood fragrance. The evaluation standard is as follows. The evaluated values are the average of 20 panels of specialist.

(Evaluation standard)

- \pm : Working example 8 (Comparison standard)
- +: The smell quality with fragrant wood was improved in comparison with Working example

8

-: The smell quality with fragrant wood was inferior in comparison with Working example 8

The result is shown in Table 6.

Table 6

Peri	ume material	Work.	Work	Comp.
		Ex.8	Ex.9	Ex.4
		150	150	150
1	Cedarwood oil	150	150	150
2	Iso E super(*1)	150	150	150
3	Phenylethyl alcohol	100	100	100
4	Hedion(*2)	100	100	100
5	Sandalore(*3)	80	80	80
6	Galaxolide(*4)	80	80	80
7	Bergamot oil	70	70	70
8	α -Isomethyl ionone	50	50	50
9	Lilial(*5)	50	50	50
10	Lyral(*6)	30	30	30
11	Eugenol	30	30	30
12	Muscone	30	30	30
13	Cypress oil	20	20	20
14	Patchouli oil	20	20	20
15	Vanillin	10	10	10
16	Rose oil	5	5	5
17	Clove oil	5	5	5
18	Oakmoss absolute	5	5	5
19	Cinnamon bark oil	3	3	3
20	Ylang ylang oil	2	2	2
21	Fragrance collection liquid	10	5	_
22	Fragrant wood extract	,	5	10

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Total (by weight)	1000	1000	1000
Smell quality evaluation	土	+	_

- *1 Trade name of IFF Inc.
- *2 Trade name of Firmenich Inc.
- *3 Trade name of Givaudan-Roure Inc.
- *4 Trade name of IFF Inc.
- *5 Trade name of Givaudan-Roure Inc.
- *6 Trade name of IFF Inc.

(Manufacturing method of fragrant wood extract)

Chips of agarwood ("Kyara") was kept to stand for 10 days in ethanol solution of 19 weight times the agarwood. After filtering, decoloring process was carried out with active carbon. The decolored solution was filtered and a crude extract of the agarwood was obtained. Under the reduced pressure the ethanol is removed by an evaporator and the fragrant wood extract was obtained.

In Table 6, it is shown that smell quality was improved by adding fragrance collection liquid and fragrant wood extract. Also, it is shown that excellent aroma is not obtained in the composition containing the fragrant wood extract only.

Next, the weight ratio of fragrance collection liquid to fragrant wood extract was studied by using composition of Table 5. The evaluation standard is as follows. The evaluated values are the average of 20 panels of specialist.

(Evaluation standard)

- 2: The smell quality was improved in comparison with Working example 8
- 1: The smell quality was improved slightly in comparison with Working example 8
- 0: The smell quality did not change in comparison with Working example 8 or the smell

quality was inferior to Working example 8

The result is shown in Table 7.

Table 7

				1.	<u></u>		
Perfume material				example		_	7
	1	2	3	4	5	6	7
Cedarwood oil	150	150	150	150	150	150	150
Iso E soper (*1)	150	150	150	150	15 0	150	150
Phenylethyl alcohol	100	100	100	100	10 0	100	100
Hedion (*2)	100	100	100	100	100	100	100
Sandalore (*3)	80	80	80	80	80	80	80
Galaxolide (*4)	80	80	80	80	80	80	80
Bergamot oil	70	70	70	70	70	70	70
α -Isomethyl ionone	50	50	50	50	50	50	50
Lilial (*5)	50	50	50	50	50	50	50
Lyral (*6)	30	30	30	30	30	30	30
Eugenol	30	30	30	30	30	30	30
Muscone	30	30	30	30	30	30	30
Cypress oil	20	20	20	20	20	20	20
Patchouli oil	20	20	20	20	20	20	20
Vanillin	10	10	10	10	10	10	10
Rose oil	5	5	5	5	5	5	5
Clove oil	5	5	5	5	5	5	5
Oakmoss absolute	5	5	5	5	5	5	5
Cinnamon bark oil	3	. 3	3	3	3	3	3
Ylang ylang oil	2	2	2	2	2	2	2
Fragrance collection liquid	1	2	3	5	8	9	0

Fragrant wood extract	9	8	7	5	2	1	10
Total (by weight)	1000	1000	1000	1000	1000	1000	1000
Ratio(*)	0.11	0.25	0.43	1.00	4.00	9.00	0.00
Evaluation	0	1	2	2	2	1 	0

^{*1} Trade name of IFF Inc.

In Table 5, it is understood that the ratio of the fragrance collection liquid/the fragrant wood extract is preferably 0.25 to 9.

The working example of perfume composition for the perfume is shown in Table 8.

Table 8

200	
150	
150	
100	
100	
80	
	150 150 100 100

^{*2} Trade name of Firmenich Inc.

^{*3} Trade name of Givaudan-Roure Inc.

^{*4} Trade name of IFF Inc.

^{*5} Trade name of Givaudan-Roure Inc.

^{*6} Trade name of IFF Inc.

^{*} Ratio = Fragrance collection liquid/Fragrant wood extract

WO 00/78117 PCT/JP00/03522

Hexyl cinnamic aldehyde	50		
Geraniol	30		
Eugenol	30		
Habanolide(*5)	30		
Bergamot oil	20		
Processed-valerian	20 .		
Muscone	10		
Jasmine oil	5		
Vanillin	5		
Rose oil	3		
Tonka beans absolute	3		
Benzoin Resinoid	3		
Oakmoss absolute	3		
Helional(*6)	2		
Patchouli oil	2		
Ambrette seed oil	1		
Orris concrete	1		
Fragrance collection liquid	1		
Tatal (by weight)	1000		_

^{*1} Trade name of IFF Inc.

As explained with the working examples of the present invention, a method of

^{*2} Trade name of Firmenich Inc.

^{*3} Trade name of Givaudan-Roure Inc.

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^{*5} Trade name of Firmenich Inc.

^{*6} Trade name of IFF Inc.

collection of fragrance ingredient of the present invention comprises solvent extraction of an essential oil ingredient from a fragrant wood and collection of the fragrance ingredient in vapor-phase obtained by heating the extract. Accordingly, because the fragrance ingredient obtained has not burnt smell at all, it can be utilized as material for various uses. Only the fragrance part of the fragrant wood can be utilized fully. Namely, without being limited to conventional incense, incense stick etc., the application and the utilization of the wide area of perfume, cosmetics, aromatic etc. becomes possible.

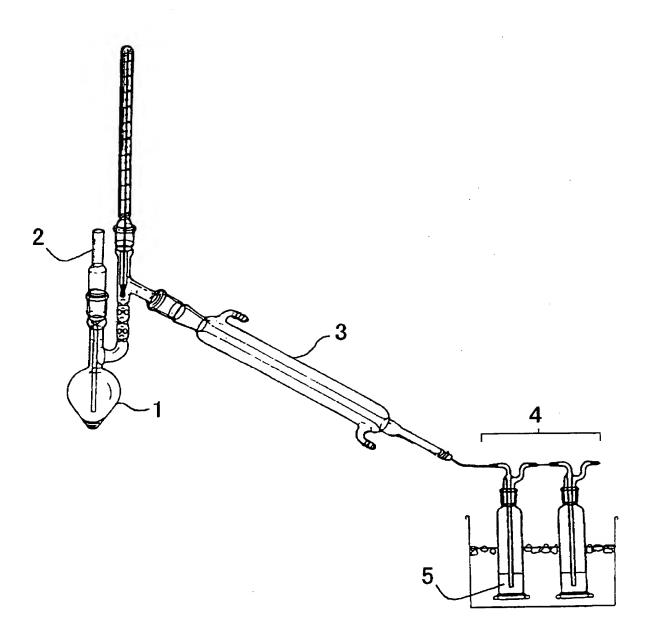
Also, by adding the fragrant wood extract to the fragrance ingredient of the present invention, an aroma quality can be improved. The fragrance of fragrant wood can be reproduce more accurately.

CLAIMS

- 1. A method of collection of a fragrance ingredient with comprises extracting an essential oil ingredient by solvent from a fragrant wood and collecting the fragrance ingredient in vapor-phase by heating the extract
- 2. The method of collection of a fragrance ingredient according to claim 1, wherein the fragrance ingredient is discharged forcibly from a heating container by introducing inert gas.
- 3. The method of collection of the fragrance ingredient according to claim 1 or 2, wherein the fragrance ingredient is collected to absorption solvent and get a resultant as a fragrance collection liquid.
- 4. The method of collection of the fragrance ingredient according to any one of claims 1 to 3, wherein the fragrant wood is agarwood.
- 5. A perfume composition containing the fragrance ingredient by means of the method of collection of the fragrance ingredient according to any one of claims 1 to 4
- 6. The perfume composition according to claim 5, wherein the perfume composition contains a fragrant wood extract.
- 7. The perfume composition according to claim 6, wherein a ratio by weight of the fragrance collection liquid and the fragrant wood extract(fragrance collection liquid/ fragrance wood extract) is 0.25 to 9.
- 8. The perfume composition according to any one of claims 5 to 7, wherein said perfume composition has an aroma of agarwood.
- 9. A cosmetic containing the fragrance ingredient by means of the method of collection of the fragrance ingredient according to any one of claims 1 to 4
- 10. The cosmetic according to claim 9, wherein said cosmetic contains a fragrant wood extract.
- 11. The cosmetic according to claim 10, wherein a ratio by weight of the fragrance collection liquid and the fragrant wood extract(fragrance collection liquid/ fragrant wood extract) is 0.25 to 9.

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FIG. 1



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